

REPORT DOCUMENTATION PAGE

AFRL-SR-BL-TR-98-

0230

Public reporting burden for this collection of information is estimated to average 1 hour per response, including gathering and maintaining the data needed, and completing and reviewing the collection of information. Send collection of information, including suggestions for reducing this burden, to Washington Headquarters Service, Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork

ces.
this
rson

1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE		3. REPORT TYPE AND DATES COVERED FINAL 01 Jul 94 To 31 Dec 97	
4. TITLE AND SUBTITLE AASERT-94 CHEMISTRY INVOLVING THE PREPARATION, ISOLATION, AND IMMOBILIZATION OF NANOCRYSTALLINE AND/OR MICROCRYSTALLINE BORON ARSENIDE, BORON PHOSPHIDE, & BORON ANTIMONIDE				5. FUNDING NUMBERS F49620-94-1-0324 3484/XS 61103D	
6. AUTHOR(S) Dr Richard L. Wells					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Dept of Chemistry Duke University P.O. Box 90346 Durham NC 27708-0346				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) AFOSR/NL 110 Duncan Ave Room B115 Bolling AFB DC 20332-8050 Maj Hugh C. De Long				10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES					
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.					
12b. DISTRIBUTION STATEMENT DTIC QUALITY INSPECTED 2					
13. ABSTRACT (Maximum 200 words) During this report period, publications 1 and 2 (see below) appeared in print (the contents of the papers were reported in two previous "Interim Technical Reports"). Michael Lube prepared the adduct Cl3B.Sb(Sb(SiMe3)3, the third Lewis acid-base adduct of boron and antimony to be structurally characterized. This adduct along with Br3 B.Sb(SiMe3)3 and I3B.Sb(SiMe3)3 are the subject of publication 3 (see below) with the manuscript being written by Michael. Based on the various data obtained, it appeared that the black powders obtained from the thermolysis of these adducts were a mixture of nanocrystalline Sb(hex) and amorphous BSb (data summarized in Michael's PhD. dissertation, 9/6/96, Duke University). Further investigations, on halo-boron-arsenic systems by Michael, with some assistance from Richard Jouet, (see publication 4 below) resulted in the isolation and structural characterization of I3B.As(SiMe3)3, as well as the isolation of X-ray quality crystals of the Cl3B.As(SiMe3)3 and [I2BAS(SiMe3)2]2 (preparations previously reported in the 1996 "Interim Technical report"). Additional studies by Richard involving (Et2O)2Li(U-P(SiMe3)2]2GaH2 showed that (1) in a 1:1 mole ratio reaction with BCl3, both known [Cl2GaP(SiMe3)2]3 and new Cl2Ga[U-P(SiMe3)2BH2] were produced, (2) from a 1:1.25 mole ratio reaction with					
14. SUBJECT TERMS				15. NUMBER OF PAGES	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT (U)		18. SECURITY CLASSIFICATION OF THIS PAGE (U)		19. SECURITY CLASSIFICATION OF ABSTRACT (U)	
				20. LIMITATION OF ABSTRACT (UL)	

CONTINUE PAGE 2,

F49620-94-1-0324, Dr Well, Duke University

MeBCl₂, both (Me₃Si)₂P(H)Ga[U-P(SiMe₃)₂]₂GaH₂ and (Me₃Si)₂P(H)Ga[U-P(SiMe₃)₂]₂B(H)Me could be isolated (cocrystallized in a 1:1 mole ratio), and (3) the 1:2.8 mole ratio reaction with MeBCl₂ afforded (Me₃Si)₂P(Cl)Ga[U-P(SiMe₃)₂]₂B(H)Me as the only species to crystallize from solution. In addition, he found that the known trimer (H₂GaP(SiMe₃)₂)₃ was the only compound that could be isolated and characterized from the 1:1 mole ratio reactions of (Et₂O)₂Li)U-P(SiMe₃)₂]₂GaHe with (1) Me₂BBr and (2) H₂BCl.SMe₂.

Final Technical Report

AFOSR AASERT Grant Number: F49620-94-1-0324

Research Title: *Chemistry Involving the Preparation, Isolation, and Immobilization of Nanocrystalline and/or Microcrystalline Boron Arsenide, Boron Phosphide, and Boron Antimonide*

Period Covered: 01 July 1996 - 31 Dec. 1997 (no-cost extension for the period 01 July 1997 - 31 Dec. 1997)

Principal Investigator: Professor Richard L. Wells, Department of Chemistry, Box 90346
Duke University Durham, NC 27708-0346

Graduate Students Supported: Michael S. Lube, July 1994-Sept. 1996 (completed requirements for the Ph.D. degree September 6, 1996; currently employed by IBM)

Richard J. Jouet, July 1996-Dec. 1997 (third-year graduate student; grades satisfactory; passed Ph.D. preliminary examination March 10, 1997)

Brief Narrative Report of Research Results: During this report period, publications 1 and 2 (see below) appeared in print (the contents of the papers were reported in two previous "Interim Technical Reports"). Michael Lube prepared the adduct $\text{Cl}_3\text{B}\cdot\text{Sb}(\text{SiMe}_3)_3$, the third Lewis acid-base adduct of boron and antimony to be structurally characterized. This adduct along with $\text{Br}_3\text{B}\cdot\text{Sb}(\text{SiMe}_3)_3$ and $\text{I}_3\text{B}\cdot\text{Sb}(\text{SiMe}_3)_3$ are the subject of publication 3 (see below), with the manuscript being written by Michael. Based on the various data obtained, it appeared that the black powders obtained from the thermolysis of these three adducts were a mixture of nanocrystalline Sb(hex) and amorphous BSb (data summarized in Michael's Ph.D. dissertation, 9/6/96, Duke University). Further investigations on halo-boron-arsenic systems by Michael, with some assistance from Richard Jouet, (see publication 4 below) resulted in the isolation and structural characterization of $\text{I}_3\text{B}\cdot\text{As}(\text{SiMe}_3)_3$, as well as the isolation of X-ray quality crystals of the $\text{Cl}_3\text{B}\cdot\text{As}(\text{SiMe}_3)_3$ and $[\text{I}_2\text{BAS}(\text{SiMe}_3)_2]_2$ (preparations previously reported in the 1996 "Interim Technical Report"). Additional studies by Richard involving $(\text{Et}_2\text{O})_2\text{Li}(\mu\text{-P}(\text{SiMe}_3)_2)_2\text{GaH}_2$ showed that (1) in a 1:1 mole ratio reaction with BCl_3 , both known $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_3$ and new $\text{Cl}_2\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{BH}_2$ were produced, (2) from a 1:1.25 mole ratio reaction with MeBCl_2 , both $(\text{Me}_3\text{Si})_2\text{P}(\text{H})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{GaH}_2$ and $(\text{Me}_3\text{Si})_2\text{P}(\text{H})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{B}(\text{H})\text{Me}$ could be isolated (cocrystallized in a 1:1 mole ratio), and (3) the 1:2.8 mole ratio reaction with MeBCl_2 afforded $(\text{Me}_3\text{Si})_2\text{P}(\text{Cl})\text{Ga}[\mu\text{-P}(\text{SiMe}_3)_2]_2\text{B}(\text{H})\text{Me}$ as the only species to crystallize from solution. In addition, he found that the known trimer $[\text{H}_2\text{GaP}(\text{SiMe}_3)_2]_3$ was the only compound that could be isolated and characterized from the 1:1 mole ratio reactions of $(\text{Et}_2\text{O})_2\text{Li}(\mu\text{-P}(\text{SiMe}_3)_2)_2\text{GaH}_2$ with (1) Me_2BBr and (2) $\text{H}_2\text{BCl}\cdot\text{SMe}_2$.

Publications Describing Results Obtained

1. M. S. Lube, R. L. Wells, and P. S. White, "Preparation and Characterization of Halogen-Boron-Phosphorus Compounds; X-ray Crystal Structures of $[\text{X}_3\text{B}\cdot\text{P}(\text{SiMe}_3)_3]$ and $[\text{X}_2\text{BP}(\text{SiMe}_3)_2]_2$ ($\text{X} = \text{Cl}, \text{Br}$)", *Inorg. Chem.* **1996**, *35*, 5007.
2. M. S. Lube, R. L. Wells, and P. S. White, "Reactions of Boron Trihalides with Tris(trimethylsilyl)arsine and Lithium Bis(trimethylsilyl)arsenide; X-ray Crystal Structures of $[\text{X}_2\text{BAS}(\text{SiMe}_3)_2]_2$ ($\text{X} = \text{Cl}, \text{Br}$)", *Main Group Metal Chemistry* **1996**, *19*, 733. (invited manuscript)
3. M. S. Lube, R. L. Wells, and P. S. White, "Synthesis, Characterization, and X-ray Crystal Structures of the Boron-Antimony adducts $\text{X}_3\text{B}\cdot\text{Sb}(\text{SiMe}_3)_3$ ($\text{X} = \text{Cl}, \text{Br}, \text{I}$)", *J. Chem Soc., Dalton Transactions* **1997**, 285.
4. M. S. Lube, R. J. Jouet, R. L. Wells, P. S. White, and V.G. Young, Jr., "Further Investigations into the Synthesis and Characterization of Halo-Boron-Arsenic Compounds: X-ray Crystal Structures of $\text{X}_3\text{B}\cdot\text{As}(\text{SiMe}_3)_3$ ($\text{X} = \text{Cl}, \text{I}$) and $[\text{I}_2\text{BAS}(\text{SiMe}_3)_2]_2$ ", *Main Group Chemistry* in press.

Presentations at Meetings Describing Results Obtained

1. M. S. Lube, R. L. Wells, and P. S. White. "Syntheses, Characterization and Thermolyses of Potential Precursors to Boron Antimonide." 212th National Meeting of the American Chemical Society, August 25-29 1996, Orlando, FL.
2. R. J. Jouet, R. L. Wells, and A. L. Rheingold, "Investigations into the Reactivity of the Lithium Pnictidogallates: Reactions of $(\text{Et}_2\text{O})_2\text{LiE}(\text{SiMe}_3)_2\text{Ga}(\text{H})_2\text{E}(\text{SiMe}_3)_2$ ($\text{E} = \text{P}, \text{As}$) with BCl_3 and R_2BCl ($\text{R} = \text{H}, \text{Me}$)", 111th Sectional Conference of the North Carolina Section of the American Chemical Society, Durham, NC, April 5, 1997. (* should have read "..... with BCl_3 , $\text{H}_2\text{BCl}\cdot\text{SMe}_2$ and MeBCl_2 ")
3. R. J. Jouet, R. L. Wells, J. F. Janik, and P. S. White, "Lithium Pnictidogallate Reactivity: Reactions of $(\text{Et}_2\text{O})_2\text{Li}[\mu\text{-E}(\text{SiMe}_3)_2]_2\text{GaH}_2$ ($\text{E} = \text{P}, \text{As}$) with Group 13 Halides", Abstracts of Papers, 214th American Chemical Society National Meeting, September 7-11, 1997, INOR 351, Las Vegas, NV.